Synthesis and photochromic properties of fulgides and fulgimides, 5-alkoxybenzo[*b*]furan derivatives*

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New heterocyclic fulgides, 3-[1-(5-alkoxy-2-methylbenzo[*b*]furan-3-yl)ethylidene]-4-(2-propylidene)dihydro-2,5-furandiones, and fulgimides, 1-(2-dimethylaminoethyl)-3-[(5-methoxy-2-methylbenzo[*b*]furan-3-yl)ethylidene]-4-(2-propylidene)dihydro-2,5-pyrroledione and 1-benzyl-3-[(5-methoxy-2-methylbenzo[*b*]furan-3-yl)ethylidene]-4-(2-propylidene)dihydro-2,5-pyrroledione, were synthesized. Electron spectroscopy and ^{1}H NMR spectroscopy were used to establish that all the compounds obtained have *Z*-configuration and exhibit photochromic properties with high stability to photodegradation in solutions. Cyclic photoisomers of fulgides possess fluorescent properties and thermal stability.

Key words: benzo[*b*]furan, fulgide, fulgimide, synthesis, photochromism.

Bistable photochromic compounds, among which heterocyclic fulgides and fulgimides are the most promising, attract attention as materials for molecular electronics devices,[1] since they are characterized by thermal stability of both the initial and the photoinduced forms and high resistance to photodegradation.[2—6]

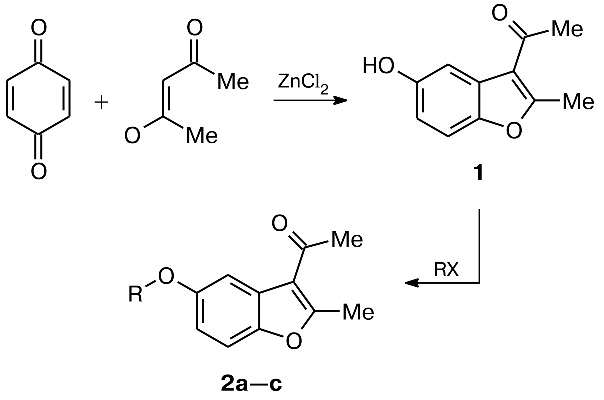
The practical use of these molecular switches is broadened due to the presence of additional properties, such as fluorescence of the initial[7] or the photo form.[8,9] A possibility to form magnetoactive[10] or supramolecular structures,[11] including nanoaggregates and Langmuir—Blodgett films, is also important.[12]

In the present work, we synthesized new (5-alkoxybenzo[*b*]furan-3-yl)fulgides and (5-methoxybenzo[*b*]-furan-3-yl)fulgimides in order to search for molecular switches with the fluorescent colored form.

Results and Discussion

The condensation of 1,4-benzoquinone and acetylacetone leads to 3-acetyl-5-hydroxy-2-methylbenzo[*b*]furan (**1**). The alkylation of the sodium salt of the latter with alkyl halides in ethanol or dimethyl sulfate in dioxane gives 3-acetyl-5-alkoxy-2-methylbenzo[*b*]furans **2a—c** (Scheme 1).

Scheme 1



R = Me (**a**), C_6H_{13} (**b**), $C_{16}H_{33}$ (**c**)

The Stobbe condensation of acetylbenzofurans **2a—c** with diethyl isopropylidenesuccinate in THF in the presence of sodium hydride led to the corresponding monoesters of substituted diethylidenesuccinic acids **3a—c**, hydrolysis of which with alcoholic alkali and subsequent cyclization of diacids **4a—c** assisted by acetic anhydride leads to fulgides **5a—c** (Scheme 2).

Fulgimides **6a**,**b** were synthesized by the reaction of fulgide **5a** with amines and subsequent cyclization using hexamethyldisilazane with zinc chloride[13] (Scheme 3).

The structures of fulgides **5a—c** and fulgimides **6a**,**b** were established by IR spectroscopy and ^{1}H NMR spectroscopy.

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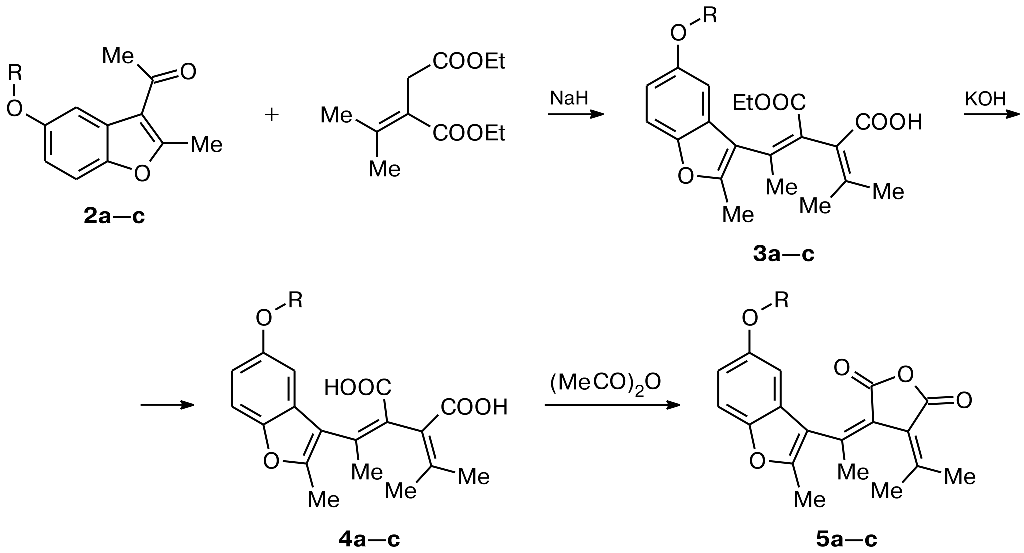
Scheme 2

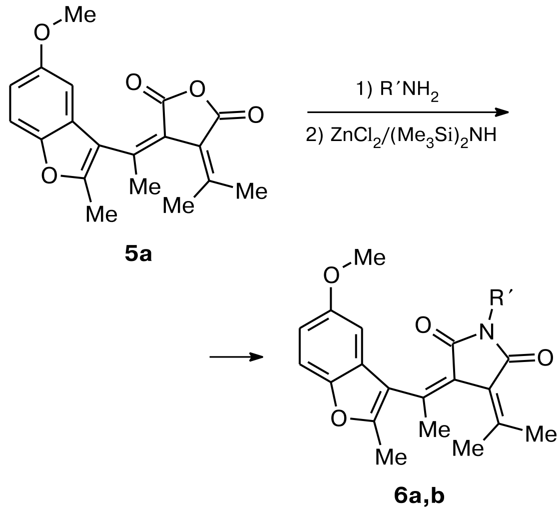


The IR spectra of fulgides **5a—c** exhibit characteristic vibration bands of two exocyclic carbonyl groups at 1786—1749 and 1818—1799 cm^{-1} attributable to the substituted diethylidenesuccinic anhydride. Their ^{1}H NMR spectra contain high-field signals of the alkoxy group and four alkyl groups and low-field signals of three aromatic hydrogen atoms. The absence in the ^{1}H NMR spectrum of the signals of one of the methyls of the isopropylidene fragment in the region δ 1 indicates the *Z*-configuration of fulgides **5a—c** with respect to the heterylethylidene double bond structurally unprepared to the photo-initiated electrocyclic reaction.[4]

The IR spectra of fulgimides **6a** and **6b** exhibit characteristic vibration bands of the atoms of the C=O valent bonds of the pyrrole-2,5-dione ring in the regions 1700, 1691 cm^{-1} and 1750, 1747 cm^{-1}, respectively, that confirms the presence of substituted diethylidenesuccinylimide fragment.

The ^{1}H NMR spectra of solutions of fulgimides of the benzo[*b*]furan series **6a**,**b** in deuterochloroform exhibit the high-field four three-proton singlets (two isopropylidene methyls and two methyl groups of the benzofuranylethylidene fragment), a three-proton singlet of the methoxy group (δ ~3.8.), and multiplets of substituents at the nitrogen atom of the pyrroledione fragment: for fulgimide **6a**, these are two three-proton multiplets and a six-proton singlet of the dimethylamino group; for fulgimide **6b**, two doublets (AB-system) of the benzyl methylene group are observed in the region δ 4.6. Signals of three aromatic protons of the benzofuran fragment are found in the low-field of these spectra in the region δ 6.7—7.3. The absence of the signals for the methyl group of the isopropylidene fragment in the region δ 1, like in the case of fulgides, indicates the *Z*-configuration of the synthesized fulgimides **6a**,**b**. The presence of the AB-system of the benzyl methylene protons indicates chirality of these molecules with a high barrier of racemization.

Scheme 3

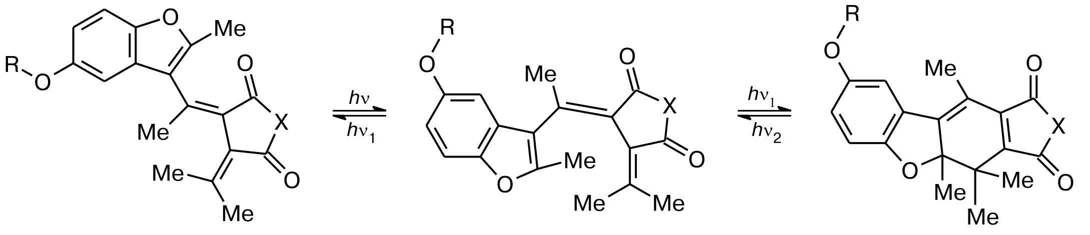


R′ = $CH_2CH_2NMe_2$ (**a**), CH_2Ph (**b**)

The electron absorption spectra (EAS) of solutions of fulgides **5a—c** and fulgimides **6a**,**b** in toluene (*Z*-isomer, Scheme 4) are characterized by the presence of the long-wavelength absorption bands with the maxima at 344 (**5a**), 346 (**5b**,**c**), 326 (**6a**), 330 nm (**6b**) and the molar extinction coefficients in the maxima of 9300, 9200, 9150, 7170, and 8230 L mol^{-1} cm^{-1}, respectively. These compounds do not possess fluorescent properties at ~20 °C.

When irradiated with UV light (365 nm), solutions of fulgides **5a—c** and fulgimides **6a**,**b** acquire color, which is

Scheme 4



Z-**5**, *Z*-**6** *E*-**5**, *E*-**6** *C*-**5**, *C*-**6**

5: X = O, R = Me (**a**), C_6H_{13} (**b**), $C_{16}H_{33}$ (**c**)
6: X = NR′, R = Me, R′ = $CH_2CH_2NMe_2$ (**a**), CH_2Ph (**b**)

accompanied by the appearance of the bands with the maxima at 500 (**5a—c**), 488 (**6a**), or 494 nm (**6b**) in the visible region of the EAS characteristic of cyclic isomers *C* (see Scheme 4).[5,9,14] A typical transformation of EAS of fulgides **5** and fulgimides **6** upon exposure to UV light is shown in Fig. 1 for fulgide **5c** as an example.

The cyclic forms *C* of fulgides **5** and fulgimides **6** possess high thermal stability: at ~20 °C, the reverse dark reaction $C \to E$ was not observed over 48 h.

Unlike fulgimides **6**, cyclic isomers *C* of fulgides under study in solutions in toluene possess a weak fluorescence with the maxima at 603 (**5a**) and 608 nm (**5b**,**c**) (see Fig. 1).

The exposure of the colored solutions to visible light (546 nm) causes their bleaching as a result of the reverse ring opening photoreaction $C \to E$.

A prolonged UV irradiation of fulgides and fulgimides does not lead to a complete conversion to the colored form because of the overlap of absorption bands $S_0 \to S_2$ of cyclic isomers *C* with absorption bands of the $S_0 \to S_1$ transition of the open *E*- and *Z*-isomers.[5,14] Since there are a reverse recyclization photoreaction $C \to E$ and *Z*/*E*-photoisomerization characteristic of fulgides and fulgimides, a photostationary state is reached after UV irradiation, which includes *E*-, *Z*-, and *C*-isomeric forms. A subsequent full photobleaching of the solution with visible light leads to a mixture of only *E*- and *Z*-isomers, and the initial spectrum (*Z*-isomer) is not restored after the bleaching.

Fulgides **5a—c** and fulgimides **6a**,**b** are characterized by stability to photodegradation. Upon repetition of ten cycles photocoloring—photobleaching of the toluene solutions of these compounds, for example, compound **6b** (Fig. 2), no decrease of optical density in the maximum of the long-wavelength absorption band of cyclic *C*-form in the photostationary state was observed.

In conclusion, we synthesized new photochromic (5-alkoxybenzo[*b*]furan-3-yl)fulgides and (5-methoxy-2-methylbenzo[*b*]furan-3-yl)fulgimides, which are characterized by thermal stability of photoinduced cyclic forms. Fulgides, as compared to fulgimides, possess more long-wavelength absorption by both the initial (*Z*) and the photoinduced cyclic (*C*) isomeric forms. Variation of alkyl

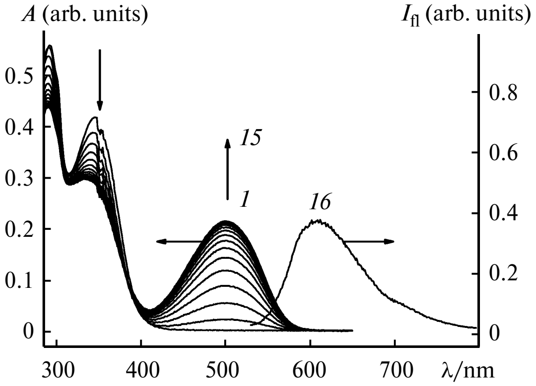


Fig. 1. Electron absorption spectra (*1—15*) of a solution of fulgide **5c** in toluene ($c = 4.5 \cdot 10^{-5}$ mol L^{-1}, l = 1 cm, T = 293 K) upon exposure to the light with λ = 365 nm recorded every 1 min and fluorescence spectrum (λ_{ex} = 500 nm) (*16*) of the cyclic *C*-isomer.

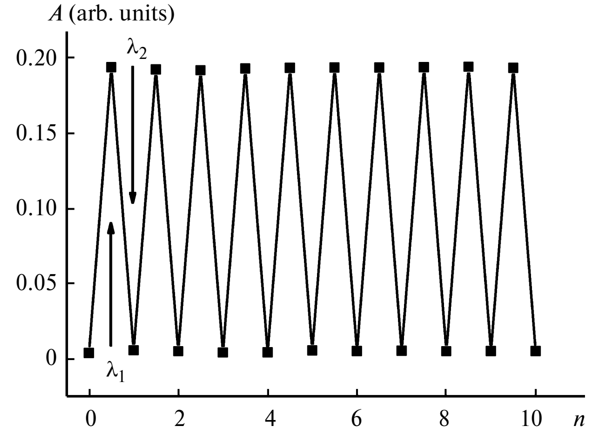


Fig. 2. Changes in the optical density of a toluene solution ($c = 4.5 \cdot 10^{-5}$ mol L^{-1}, l = 1 cm, T = 293 K) of fulgimide **6b** in the absorption maximum of the *C*-form (494 nm) upon repetition of the coloring (exposure to the light with λ_1 = 365 nm for 18 min)—bleaching (exposure to the light with λ_2 = 546 nm for 24 min) cycles; *n* is the number cycles.

substituent in the 5-alkoxy group virtually has no effect on the photochromic and fluorescent properties of fulgides synthesized. Thermal stability and luminescent properties of the colored form of fulgides allow us to consider them as molecular switches with fluorescent signal function.

Experimental

IR spectra were recorded on a Varian Excalibur 3100 FT-IR spectrometer, ^{1}H NMR spectra were recorded on a Varian Unity-300 spectrometer (300 MHz) in $CDCl_3$, using residual signals of $CHCl_3$ (δ 7.25) as the reference. Electron absorption spectra were obtained on a Cary 100 spectrophotometer (Varian), fluorescence spectra were obtained on a Varian Eclipse spectrofluorimeter. Solutions under study were exposed to the radiation of a DRSh-250 mercury lamp with a kit of interference light filters for selection of the mercury spectrum lines in a quartz cell (*l* = 1 cm).

3-Acetyl-5-hydroxy-2-methylbenzo[*b*]furan (1). Anhydrous $ZnCl_2$ (45 g, 0.33 mol), anhydrous ethanol (57 mL), and acetylacetone (100 mL, 1 mol) were placed into a 250-mL three-neck flask equipped with a thermometer, a reflux condenser with a calcium chloride drying tube, and a stirrer. The mixture was heated with stirring using a boiling water bath until the salt was dissolved, followed by addition (at the same temperature) in portions of 1,4-benzoquinone (35.5 g, 0.33 mol) and heating in the water bath for 40 min. Then, the mixture was cooled, a precipitate formed was filtered off, washed with cold ethanol, dried in air, and recrystallized from ethanol. The yield was 61%, m.p. 240 °C with decomp. (*cf.* Ref. 14: m.p. 234—235 °C).

3-Acetyl-5-methoxy-2-methylbenzo[*b*]furan (2a). A mixture of compound **1** (19.02 g, 0.1 mol), 2 *M* solution of NaOH (100 mL), dioxane (50 mL), and dimethyl sulfate (20.8 mL, 0.22 mol) was stirred for 2 h at ~20 °C. Then, the reaction mixture was poured into ice-cold water (150 mL) and was allowed to stand for 12 h in a refrigerator. A precipitate formed was filtered off, washed with cold water until neutrality, dried in air, and recrystallized from butanol. The yield was 17.3 g (81%), m.p. 90 °C (*cf.* Ref. 15: m.p. 44—45 °C). IR, ν/cm^{-1}: 1651 (C=O). ^{1}H NMR ($CDCl_3$), δ: 2.59, 2.74 (both s, 3 H each, Me); 3.86 (s, 3 H, OMe); 6.86 (dd, 1 H, CH arom., J_1 = 9.0 Hz, J_2 = 2.7 Hz); 7.30 (d, 1 H, CH arom., *J* = 9.0 Hz); 7.43 (d, 1 H, CH arom., *J* = 2.7 Hz).

3-Acetyl-5-hexyloxy-2-methylbenzo[*b*]furan (2b). Compound **1** (3.8 g, 0.02 mol) and *n*-hexyl iodide (3.24 mL, 0.022 mol) were sequentially added to a solution of sodium ethylate prepared from Na (0.58 g, 0.025 mol) and anhydrous ethanol (15 mL). The reaction mixture was refluxed for 30 min (a precipitate partially dissolved), cooled, and diluted with water (250 mL). An oil formed was extracted with diethyl ether, the ether was evaporated, and the residue was purified by column chromatography (silica gel, chloroform). A colorless oil of hexyloxybenzofuran **2b** obtained after evaporation of the solvent was crystallized by treatment with light petroleum. The yield was 3.83 g (70%), colorless crystals (from butanol), m.p. 52—53 °C. Found (%): C, 74.23; H, 8.14. $C_{17}H_{22}O_3$. Calculated (%): C, 74.42; H, 8.08. IR, ν/cm^{-1}: 1664 (C=O). ^{1}H NMR ($CDCl_3$), δ: 0.85—0.95 (m, 3 H, Me); 1.25—1.85 (m, 8 H, CH_2); 2.59, 2.74 (both s, 3 H each, Me); 3.99 (t, 2 H, CH_2, *J* = 6.6 Hz); 6.86 (dd, 1 H, CH arom., J_1 = 8.7 Hz, J_2 = 2.4 Hz); 7.29 (d, 1 H, CH arom., *J* = 8.7 Hz); 7.43 (d, 1 H, CH arom., *J* = 2.4 Hz).

3-Acetyl-5-hexadecyloxy-2-methylbenzo[*b*]furan (2c) was obtained similarly to **2b** from compound **1** and 1-bromohexadecane. The yield was 55%, m.p. 85 °C (from ethanol). Found (%): C, 78.33; H, 10.17. $C_{27}H_{42}O_3$. Calculated (%): C, 78.21; H, 10.21. IR, ν/cm^{-1}: 1665 (C=O). ^{1}H NMR ($CDCl_3$), δ: 0.85—0.95 (m, 3 H, Me); 1.10—1.85 (m, 28 H, CH_2); 2.59, 2.74 (both s, 3 H each, Me); 3.99 (t, 2 H, CH_2, *J* = 6.6 Hz); 6.86 (dd, 1 H, CH arom., J_1 = 8,7 Hz, J_2 = 2.4 Hz); 7.29 (d, 1 H, CH arom., *J* = 8.7 Hz); 7.43 (d, 1 H, CH arom., *J* = 2.4 Hz).

(2*Z*)-2-[1-(5-Methoxy-2-methylbenzo[*b*]furan-3-yl)ethylidene]-3-(2-propylidene)butanedioic acid (4a). A solution of benzofuran **2a** (2.04 g, 0.01 mol) and diethyl isopropylidenesuccinate (2.8 g, 0.013 mol) in THF (20 mL) was added to a suspension of NaH (0.7 g, 0.03 mol) in THF (10 mL). After a drop of methanol was added, a characteristic crimson color appeared and evolution of hydrogen began. The reaction mixture was allowed to stand until hydrogen stopped to evolve and then for additional 15 h. The solution, which acquired a brown color, was diluted with water (170 mL), impurities were extracted with diethyl ether. The aqueous layer was acidified with 10% aqueous HCl to pH 1. An oil formed was extracted with diethyl ether. The ether was evaporated on a rotary evaporator. Monoethyl ester **3a**, obtained as an oil, was refluxed for 4 h with 10% KOH in methanol (20 mL). After evaporation of methanol, the reaction mixture was diluted with water to the volume of 200 mL and acidified with 10% aq. HCl to pH 1. A precipitate of diacid **4a** was filtered off, washed with methanol, and recrystallized from toluene. The yield was 1.6 g (46.5%), m.p. 240 °C (with decomp.). Found (%): C, 66.39; H, 5.66. $C_{19}H_{20}O_6$. Calculated (%): C, 66.27; H, 5.8. IR, ν/cm^{-1}: 3300—2400 (OH), 1685 (C=O). ^{1}H NMR ($CDCl_3$), δ: 1.93, 1.97, 2.23, 2.33 (all s, 3 H each, Me); 3.99 (s, 3 H, OMe); 6.74 (m, 2 H, CH arom.); 7.20 (m, 1 H, CH arom.).

(3*Z*)-3-[1-(5-Methoxy-2-methylbenzo[*b*]furan-3-yl)ethylidene]-4-(2-propylidene)dihydro-2,5-furandione (5a). A solution of diacid **4a** (1.6 g, 4.7 mmol) in acetic anhydride (1 mL) was refluxed for 5 min and cooled. Crystals of **5a** were filtered off, washed with methanol, and recrystallized. The yield was 63%, m.p. 164 °C, colorless crystals (from butanol). Found (%): C, 69.70; H, 5.37. $C_{19}H_{18}O_5$. Calculated (%): C, 69.93; H, 5.56. IR, ν/cm^{-1}: 1810, 1756 (C=O). ^{1}H NMR ($CDCl_3$), δ: 2.04, 2.28, 2.36, 2.46 (all s, 3 H each, Me); 3.78 (s, 3 H, OMe); 6.77—6.85 (m, 2 H, CH arom.); 7.32 (m, 1 H, CH arom.).

(2*Z*)-2-[1-(5-Hexyloxy-2-methylbenzo[*b*]furan-3-yl)ethylidene]-3-(2-propylidene)butanedioic acid (4b) was obtained similarly to acid **4a**. The yield was 11.4%, m.p. 205 °C (from toluene). Found (%): C, 69.41; H, 7.18. $C_{24}H_{30}O_6$. Calculated (%): C, 69.55; H, 7.30. IR, ν/cm^{-1}: 3300—2400 (OH), 1685 (C=O). ^{1}H NMR ($CDCl_3$), δ: 0.80—1.00 (m, 3 H, Me); 1.20—2.00 (m, 8 H, CH_2); 1.93, 1.97, 2.23, 2.33 (all s, 3 H each, Me); 3.90 (t, 2 H, OCH_2, *J* = 6.0 Hz); 6.64—6.80 (m, 2 H, CH arom.); 7.20 (m, 1 H, CH arom.); 11.5—12.1 (br.s, 2 H, OH).

(3*Z*)-3-[1-(5-Hexyloxy-2-methylbenzo[*b*]furan-3-yl)ethylidene]-4-(2-propylidene)dihydro-2,5-furandione (5b) was obtained similarly to compound **5a.** The yield was 82%, colorless crystals, m.p. 103 °C (from butanol). Found (%): C, 72.89; H, 6.91. $C_{24}H_{28}O_5$. Calculated (%): C, 72.71; H, 7.12. IR, ν/cm^{-1}: 3300—2400 (OH), 1818—1763 (C=O). ^{1}H NMR ($CDCl_3$), δ: 0.85—0.94 (m, 3 H, Me); 1.28—1.50 (m, 6 H, CH_2); 1.68—1.81 (m, 2 H, CH_2); 2.04, 2.27, 2.35, 2.46 (all s, 3 H each, Me); 3.93 (t, 2 H, OCH_2, *J* = 6.6 Hz); 6.78—6.86 (m, 2 H, CH arom.); 7.30 (m, 1 H, CH arom.).

(2*Z*)-2-[1-(5-Hexadecyloxy-2-methylbenzo[*b*]furan-3-yl)-ethylidene]-3-(2-propylidene)butanedioic acid (4c) was obtained similarly to acid **4a** from benzofuran **2c**. The yield was 25%, colorless crystals, m.p. 174 °C (from toluene). Found (%): C, 73.44; H, 8.90. $C_{34}H_{50}O_6$. Calculated (%): C, 73.61; H, 9.08. IR, ν/cm^{-1}: 3300—2400 (OH), 1685 (C=O). ^{1}H NMR (CDCl$_3$), δ: 0.85—0.95 (m, 3 H, Me); 1.10—1.80 (m, 28 H, CH$_2$); 1.92, 1.96, 2.23, 2.33 (all s, 3 H each, Me); 3.89 (t, 2 H, OCH$_2$, J = 6.3 Hz); 6.60—6.80 (m, 2 H, CH arom.); 7.20 (m, 1 H, CH arom.); 11.5—12.1 (br.s, 2 H, OH).

(3*Z*)-3-[1-(5-Hexadecyloxy-2-methylbenzo[*b*]furan-3-yl)-ethylidene]-4-(2-propylidene)dihydro-2,5-furandione (5c) was obtained similarly to compound **5a**. The yield was 73%, colorless crystals, m.p. 80 °C (from butanol). Found (%): C, 75.91; H, 8.90. $C_{34}H_{48}O_5$. Calculated (%): C, 76.08; H, 9.01. IR, ν/cm^{-1}: 1818, 1785, 1761 (C=O). ^{1}H NMR (CDCl$_3$), δ: 0.83—0.88 (m, 3 H, Me); 1.10—1.80 (m, 28 H, CH$_2$); 2.04, 2.27, 2.35, 2.46 (all s, 3 H each, Me); 3.92 (t, 2 H, OCH$_2$, J = 6.6 Hz); 6.77—6.86 (m, 2 H, CH arom.); 7.31 (m, 1 H, CH arom.).

(3*Z*)-1-(2-Dimethylaminoethyl)-3-[(5-methoxy-2-methyl-benzo[*b*]furan-3-yl)methylidene]-4-(2-propylidene)dihydro-2,5-pyrroledione (6a). 1,1-Dimethylethylenediamine (0.184 mL, 1.7 mmol) in benzene (2 mL) was added dropwise to a solution of fulgide **5a** (0.55 g, 1.7 mmol) in benzene (8 mL) with stirring and the mixture was stirred for 1 h at ~20 °C. Then, anhydrous zinc chloride (0.235 g, 1.7 mmol) was added to the reaction mixture in one portion; the mixture was heated to 80 °C and hexamethyldisilazane (0.55 mL, 3.5 mmol) in benzene (3.5 mL) was added dropwise over 10 min with stirring. The reaction mixture was refluxed with stirring for another 6 h and cooled, followed by addition of water (20 mL) and aqueous ammonium to pH 8, then the mixture was thoroughly shaken and the organic layer was separated. The solvent was evaporated on a rotary evaporator, residual water was removed by azeotropic drying with benzene. A brown oil obtained was purified by chromatography on silica gel (eluent methanol). After evaporation of the solvent, the yield of fulgimide **6a** was 0.36 g (53.9%), colorless crystals, m.p. 99—100 °C (from methanol). Found (%): C, 69.53; H, 7.27; N, 6.89. $C_{23}H_{28}N_2O_4$. Calculated (%): C, 69.68; H, 7.12; N, 7.07. IR, ν/cm^{-1}: 1750, 1700 (C=O). ^{1}H NMR (CDCl$_3$), δ: 2.00, 2.20, 2.36, 2.45 (all s, 3 H each, Me), 2.23 (s, 6 H, NMe$_2$), 2.40—2.47 (m, 2 H, NCH$_2$); 3.55—3.63 (m, 2 H, NCH$_2$); 3.81 (s, 3 H, OMe); 6.77—6.84 (m, 2 H arom.); 7.18—7.34 (m, 1 H arom.).

(3*Z*)-1-Benzyl-3-[(5-methoxy-2-methylbenzo[*b*]furan-3-yl)methylidene]-4-(2-propylidene)dihydro-2,5-pyrroledione (6b) was obtained similarly to compound **6a** from fulgide **5b** and benzylamine. The yield was 29%, colorless crystals, m.p. 95—96 °C (from benzene). Found (%): C, 74.93; H, 5.87; N, 3.49. $C_{26}H_{25}NO_4$. Calculated (%): C, 75.16; H, 6.06; N, 3.37. IR, ν/cm^{-1}: 1747, 1691 (C=O). ^{1}H NMR (CDCl$_3$), δ: 1.96, 2.18, 2.31, 2.43 (all s, 3 H each, Me); 3.77 (s, 3 H, OMe); 4.57, 4.65 (both d, 1 H each, NCH$_2$, J = 14.1 Hz); 6.76—6.90 (m, 2 H arom.); 7.18—7.37 (m, 6 H arom.).

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